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भारतीय मानक

पैट्रोलियम तथा इसके उत्पादों के परीक्षण की पद्धति

[पी : 145]

आषवीय अवचूषण स्पेक्ट्रमदर्शी द्वारा इंधन तेल और कच्चे तेल में सोडियम, निकेल और वेनाडियम

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 145]

SODIUM, NICKEL AND VANADIUM IN FUEL OILS AND CRUDE OILS BY ATOMIC ABSORPTION SPECTROSCOPY

UDC 665·6/·7 : 665·754 : 543·422 [546·33]

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 11000

January 1994

Price Group 2

Methods of Test for Petroleum, Petroleum Products and Lubricants Sectional Committee, PCD 1

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards after the draft finalized by the Methods of Test for Petroleum, Petroleum Products and Lubricants Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This Indian Standard aligns with IP 288/74 (Reapproved 1988) 'Sodium, nickel and vanadium in fuel oils and crude oils by atomic absorption spectroscopy method' issued by Institute of Petroleum, London (UK).

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 145]

SODIUM, NICKEL AND VANADIUM IN FUEL OILS AND CRUDE OILS BY ATOMIC ABSORPTION SPECTROSCOPY

1 SCOPE

This method is intended for the determination of sodium and nickel at levels above 1 μg/g and vanadium at levels above 10 μg/g in crude oils and residual fuel oils. Difficulty may be experienced in sodium determination with some samples due to the particulate nature of the sodium compounds present, and it is recommended that an ashing technique should be used for such samples.

1.2 This standard may involve hazardous materials, operations and equipments. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 SUMMARY OF METHOD

The samples are diluted in an organic solvent, the solutions are aspirated into the flame of an atomic absorption spectrometer, and the absorption of the visible or ultra-violet resonance radiation of the element being determined is measured. An acetylene/air flame is used for sodium and nickel and in acetylene/nitrous oxide flame for vanadium. Calibration curves are prepared from measurements on standard solutions of metallo-organic compounds in the same solvent.

3 APPARATUS

3.1 Atomic Absorption Spectrometer

A suitable instrument will comprise hollow cathode lamps or other sources of resonance radiation of the elements sodium, nickel, and vanadium; a gas burner with provisions for the aspiration of organic liquids into the flame; a monochromator for selecting the desired spectral lines; and a light detector and measuring system capable of scale expansion. It is recommended that a secondary air supply be used to enable the air supply to the flame to be increased without increasing sample uptake rate. Read-out of the signal on a pen recorder as well as on a meter is recommended. The scale may be linear either in light intensity or in absorbance. The instrument must be suitable for use with organic solvents; even so it is recommended that only water is left in the drain tube overnight.

4 REAGENTS

4.1 Solvent

Consisting of 10 percent (v/v) isopropyl alcohol (*see* IS 2631 : 1976), 90 percent (v/v) white spirit conforming to the following requirements to be used for the preparation of both sample and standard solutions.

Characteristics	*Requirements*
i) Clarity	Clear, no solid matter present
ii) Undissolved water at 20°C	Absent
iii) Odour	Not darker than standard colour solution (4·8 mg of pure anhydrous potassium dicromate in 1 litre of distilled water or deionized water)
iv) Distillation 1 013 mbr	Not greater than 1 ml below 130°C Not greater than 10 ml below 145°C
Volume of condensate recovered	Not less than 90 ml below 200°C End point not above 220°C
v) Aromatic content	Less than 25 percent (v/v)
vi) Residue on evaporation	Not greater than 5 mg per 100 ml

Characteristics	*Requirements*
vii) Neutrality	Neutral (50 ml of white spirit shaken with 10 ml distilled water, the water layer shall be neutral to methyl orange)
viii) Freedom from objectionable sulphur compounds	Not more than slight tarnish of copper strip
ix) Flash point	Not flashing at 32°C

NOTE — Other suitable alternative solvents like xylene or methyl isobutyl ketone have also been found satisfactory.

4.2 Standard Solutions

Suitable pure compounds for use for standard solutions are sodium tetraphenylborate (often referred to as sodium tetraphenylboron), nickel 4-cyclohexylbutyrate, bis (1-phenyl-1, 3-butanediono) oxovanadium. Alternatively the commercial naphthenates of sodium, nickel and vanadium may be used, but in this case analysis for their metal content will be required. Standards should be kept as stock solutions of 500 mg/1 or more as dilute solutions are less stable. When making further dilutions do not dilute in a single step by a factor greater than 20.

5 SODIUM DETERMINATION

5.0 Preparation of Appparatus

5.1 Consult the manufacturer's instructions for the operation of the atomic absorption spectrometer. The present method assumes that good operating procedures are followed in using the spectrometer. Design differences between spectrometers make it impracticable to specify the required manipulations in detail here.

> *Caution* — Proper operating procedures are required for safety as well as for reliability of results. An explosion can result from flame blow-back unless the correct burner head and operating sequence are used.

5.2 For the sodium determination, fit a sodium hollow cathode lamp and set the monochromator at 589·0 nm. Make fine adjustment to wavelength setting to give maximum output. Adjust gain control to set this maximum at full scale.

5.3 Using the correct burner head for acetylene/air, set up the acetylene/air flame. Aspirate a standard sodium solution into the flame. Make adjustments to the height and angle of burner and to the acetylene flow rate to give maximum absorption.

6 CALIBRATION

6.1 Freshly prepare a set of standards by diluting the 500 mg/1 master standard with the solvent.

6.2 Aspirate these standards sequentially into the flame and record the output (or note meter deflections). Aspirate solvent alone after each standard.

6.3 Determine the net absorbance of each standard. If the spectrometer output is linear in absorbance, the net absorbance is given by the difference between the absorbance for the standard or sample solution and the absorbance for the solvent alone. If the spectrometer output is proportional to transmission (that is, to light intensity) then the net absorbance is given by $\log_{10} d_0/d_1$, where the deflections are d_0 when solvent alone is aspirated and d_1 when the standard or sample solution is aspirated. However, if scale expansion is used, the ratio of light intensities required to define absorbance is not the same as the ratio of the deflections, and the absorbance is then given by $\log_{10} [(d_0+z)/(d_1+z)]$ where z is the zero shift expressed in the same units as the deflections d_0 and d_1.

Standard solutions are required to cover the range from the lowest concentration to be covered by the method up to a concentration which gives a net absorbance of about 0·75.

6.4 Plot the net absorbance against the concentration (mg/1) of sodium in the standards to give a calibration curve.

NOTE — Calibration must be carried out each day as variations occur in the instrument behaviour. Readings may also vary over short times from such causes as build-up of deposits on the burner slot or in the nebulizer. Thus, a single standard should be aspirated from time to time during a series of samples to check whether the calibration has changed (a check after every fifth sample is recommended). The visual appearance of the flame also serves as a useful check to detect some changes in condition.

7 PROCEDURE

7.1 Prepare the sample solution. As fuel oils and crude oils frequently contain water droplets and fine solid particles, viscous samples should be heated to 50 to 60°C and all samples very vigorously agitated before taking a sample for analysis. Weigh accurately in a 100 ml volumetric flask a quantity of the sample of about 10 g. Add solvent and shake well to dissolve. Make up to 100 ml with further solvent.

NOTE — 100 ml is a greater quantity of solution than is required for the subsequent measurements and, if preferred, 25 ml of solution can be prepared containing about 2·5 g of sample.

7.2 Aspirate the sample solution and determine its absorbance, aspirating solvent alone before and after. If the sample solution gives an absorbance greater than 0·75 (that is, d_1/d_0 less than 0·18) then further dilution is required. Remove 25 ml of the sample solution by pipette and dilute this to 100 ml with further solvent. Aspirate the diluted solution and determine its absorbance.

NOTE — Some samples may give irregularity of the flame or fluctuation of the signal due to the particulate nature of the sodium compounds present. If this happens the present method is unsuitable and an ashing technique should be employed.

8 CALCULATION

8.1 Read from the calibration curve the concentration *C* corresponding to the measured absorbance.

C is the concentration of sodium in the diluted sample solution, mg/l.

8.2 Calculate the sodium content of the oil in micrograms per gram as:

$$\text{sodium, } \mu g/g = \frac{VC}{W}$$

where

W = weight of sample taken, g;

V = volume of solution that contains the above weight of oil, ml (normally V is 100, but it will be 25, if the smaller volume is used and will be multiplied by a factor if further dilution has been required in **7.2**.

9 NICKEL DETERMINATION

9.1 Repeat all the operations as described in **5** to **8** but using the following conditions: acetylene/air flame; nickel hollow cathode lamp; analytical line 352·5 nm.

NOTE — 232·0 nm and 341·5 nm lines are more sensitive lines. In case of higher sensitivity requirements these lines may be used.

10 VANADIUM DETERMINATION

10.1 Repeat all the operations as described in **5** to **8** but using the following conditions: acetylene/nitrous oxide flame; vanadium hollow cathode lamp; analytical line 318·4 nm.

CAUTION — The correct burner head for this flame must be used, and the correct procedure followed for lighting. Gas flows must be kept above the specified minimal.

NOTES

1 Adjustment of flame and height of burner are important to obtain adequate sensitivity. A slightly luminous fuel-rich flame is required and the beam should pass through the rose-pink zone of the flame. Deposits should be removed from the burner slot as soon as they cause either a ragged flame or increased noise.

Scale expansion will be required for low vanadium concentration, as the method is less sensitive to vanadium than to sodium or nickel. When the absorption due to the element measured is weak, with d_1/d_0 greater than 0·8 (absorbance less than 0·1), errors in reading the output record may be reduced by the use of scale expansion. Both sample and standards are to be examined at the same degree of scale expansion.

2 Although the method has been described above for the determination of three elements on a single sample, the sequence of operations in analysing several samples should also be considered. Aspiration of a sample to determine its absorbance is very quick. Changing wavelength setting, changing lamp, and changing flame and burner type take longer time. Thus it is most economical to make measurements at single wavelength on a series of samples and standards before changing conditions.

11 REPORT

Report the content of the appropriate metal to nearest μg/g.

12 PRECISION

12.1 The following criteria should be used for judging the acceptability of results (95 percent confidence).

12.1.1 *Repeatability, r* — Duplicate results by the same operator should be considered suspect if they differ by more than the following amounts:

$r = 0{\cdot}186\ X^{0.914}$ in the determination of sodium

$r = 0{\cdot}375\ X^{0.801}$ in the determination of nickel

$r = 0{\cdot}150\ X^{0.976}$ in the determination of vanadium

where X is the mean of the two results in μg/g.

12.1.2 *Reproducibility, R*

Results from two laboratories should not be considered suspect unless they differ by more than the following amounts:

$R = 1{\cdot}049\ X^{0.914}$ in the determination of sodium

$R = 1{\cdot}146\ X^{0.801}$ in the determination of nickel

$R = 0\ 585\ X^{0.976}$ in the determination of vanadium

where X is the mean of the two results in μg/g.

NOTE — The estimates of *R* are based on very few degrees of freedom and must therefore be regarded as tentative values.

12.1.3 The following are sample values of these expressions:

Mean, X	*Sodium*		*Nickel*		*Vanadium*	
	r	*R*	*r*	*R*	*r*	*R*
10	1·5	8·6	2·4	7·3	1·4	5·5
20	2·9	16·2	4·1	12·6	2·8	10·9
30	4·2	23·5	5·7	17·5	4·2	16·2
40	5·4	30·6	7·2	22·0	5·5	21·4
50	6·6	37·5	8·6	26·3	6·8	26·6
60	7·9	44·3	10·0	30·4	8·2	31·8
70	9·0	51·0	11·3	34·4	9·5	37·0
80	10·2	57·6	12·5	38·3	10·8	42·1
90	11·4	64·1	13·8	42·1	12·1	47·3
100	12·5	70·6	15·0	45·8	13·4	52·4

NOTE — It is anticipated that the above precision estimates can be improved as greater experience is gained of the method.

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